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Optimised co-electrodeposition of Fe–Ga alloys for maximum magnetostriction effect

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ABSTRACT

The article reports the electrochemical deposition and characterisation of a 600 nm thick Fe–Ga alloy film plated on a 20 μm thick copper cantilever. The co-electrodeposition process was optimised for the production of Fe–Ga in the ratio of 81% Fe to 19% Ga, which is known to maximize the magnetostriction (MS) effect. The foil was cut into 1 mm wide and 5 mm long cantilevers and the deflection was measured with DC co-planar magnetic field intensities ranging from 0 to 60 kA/m. The maximum strain coefficient λ was measured to be 96 ppm for a field strength range 58 kA/m. The field strain plot over exhibits a typical second order magnetically induced strain curve, as seen in other magnetostrictive materials.

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1. Introduction

Magnetostriction is defined as the ability of a material to experience mechanical strain when subject to a magnetic field. The material with the highest known strain coefficient of magnetostriction, $\lambda \sim 2000$ ppm, is currently Terfenol-D, an alloy of terbium, dysprosium and iron ($\text{Tb}_{0.3}\text{Dy}_{0.7}\text{Fe}_2$) that has poor ductility and low fracture resistance making it difficult to machine [1,2]. Galfenol, an alloy of iron and gallium ($\text{Fe}_{0.8}\text{Ga}_{0.2}$) is easily machined and offers good $\lambda \sim 275\text{--}320$ ppm at relatively low field strengths (39 kA/m) [3]. This material also exhibits high tensile strength and can be used in tension unlike Terfenol-D. However high temperature and pressure are required to manufacture this alloy. Galfenol may also be deposited by sputtering though this manufacturing technique requires expensive vacuum equipment and the deposition rate depends on the partial pressure of the inert gas [1].

Recently work has been published on a method of electrochemically depositing Galfenol [4–9]. Electrodeposition of Galfenol has many advantages over previous methods of deposition, such as low cost, high deposition rate and the ability to coat large or non-planar surfaces. This method can be used for thin film deposition

and the production of nanowires [9], demonstrating the viability of this method for large-scale production of magnetostrictive microstructures and MEMS devices [7,8].

There are several challenges associated with the electrodeposition process of galfenol films [4,6]. There are only a few pH–voltage combinations where Ga ions remain stable in aqueous solutions [6]. Other challenges include hydrogen evolution due to the high electrolyte pH that leads to gallium hydroxide and oxide being formed and included in the deposits [4–6]. A combinatorial study by McGary [6] where a Hull Cell was used to vary the current density across the cathode showed that the use of complexing agents such as sodium citrate reduces the oxygen content of the electrodeposited material. More recent work by Iselt [4] achieved significantly low oxygen content through the use of alternating potential pulses.

This work is extended in this article by optimisation of the electroplating process using a previously reported electrolyte bath composition [9]. Characterisation of the resulting deposits onto 20 μm thick copper cantilever beams reveals that two alloys, $\text{Fe}_{0.81}\text{Ga}_{0.19}$ and $\text{Fe}_{0.71}\text{Ga}_{0.29}$, that display large magnetostrictive coefficients have been successfully electro-deposited [1].

2. Plating development

Co-deposition of Fe–Ga alloys from a simple, single aqueous electrolyte involves a complex deposition mechanism comprising

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Table 1
Aqueous electrolyte for Fe–Ga electrodeposition.

Component	Concentration (M)
$\text{Ga}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$	0.06
$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	0.03
H_3BO_3	0.5
$\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$	0.15
$\text{C}_6\text{H}_8\text{O}_6$	0.04

electrochemical and chemical deposition processes [4–6]. Dense, homogeneous and oxygen free films are obtained by applying alternating potential pulses [4]. This section describes the development of the plating process and elemental characterisation of the deposits.

2.1. Method

The starting aqueous electrolytes contain 0.06 M $\text{Ga}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ and 0.03 M $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ as the Fe and Ga sources, respectively. 0.5 M boric acid and 0.15 M sodium citrate are added as buffer and 0.04 M ascorbic acid acts as an antioxidant [4,5]. The pH is adjusted to less than 3.0 with dilute H_2SO_4 to avoid gallium hydroxide precipitation. The iron sulphate concentration was reduced in subsequent experiments in order to obtain the desired Fe:Ga ratio in the electrodeposits. Table 1 summarises the initial electrolyte composition for a total volume of 50 mL.

Electrodeposition experiments were performed with 50 mL of the electrolyte in a rectangular electrolytic cell with a three-electrode system in conjunction with a DropSense μStat 400 potentiostat/galvanostat. A large surface area platinum mesh was used as the counter electrode and a Saturated Calomel Electrode (SCE, 241 mV_{SHE}) as the reference electrode. All potentials refer to the SCE. A potential pulse technique was used with an “on” deposition potential E_1 and an “off” potential E_2 that were applied for $t_1 = t_2 = 3.9$ s. E_1 is equivalent to the reduction peak voltage measured from cyclic voltammetry prior to each electroplating experiment. E_1 lies in the range between -2.05 V and -2.25 V. The “off” potential E_2 was set at -0.9 V. The current during the second step E_2 was almost zero, which effectively enables the compensation of pH and concentration gradients of the metal ions. All experiments were performed at room temperature without mechanical stirring. The copper foil was cleaned using isopropanol, acetone and deionised water. Before plating the foil was immersed in Procirc 921 micro-etch solution for 10 mins to remove the copper oxide surface layer. Oxygen from the electrolyte solution was purged by the nitrogen gas for 10 mins prior to experiments and left blowing over the solution during the experiments.

According to a previously proposed deposition mechanism [4,5], each cycle led to Fe–Ga alloy deposition in parallel with gallium hydroxide precipitation during the forward potential application E_1 and the re-dissolution of hydroxide during the reverse potential step E_2 . A dense Fe–Ga alloy deposit with low oxygen content can only be achieved when hydroxide formation and re-dissolution are eliminated. The refined electrolyte recipe, which used sodium citrate as a complexing agent for the metal ions (Fe^{2+} , Ga^{3+}), allowed a stable electrolyte to be operated at a less acidic level close to pH 3.0 as opposed to pH 2.0 found in previous literature [10–12]. At this pH level, hydrogen evolution was significantly reduced. However, the increased stability of the Ga^{3+} ions also means that the release of Ga^{3+} ions became slower, especially when compared to the fast rate of Fe electrodeposition in the co-deposition electrolyte system. As a consequence, the resultant deposition contained prevalently Fe with almost no detectable trace of Ga.

To remedy this situation, Iselt et al [4,5], increased the metal ion ratio $\text{Ga}^{3+}:\text{Fe}^{2+}$ to more than 2:1 in order to enhance the reactivity

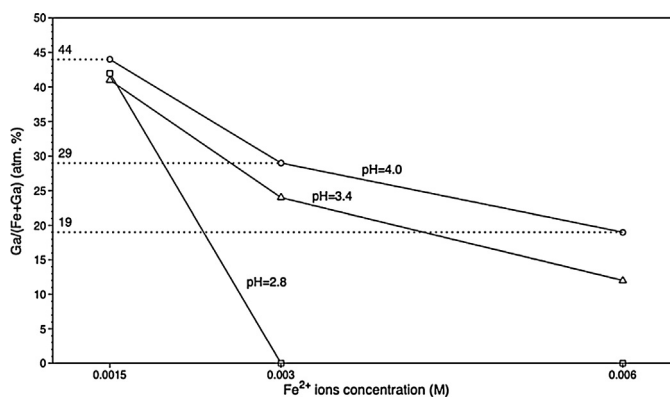


Fig. 1. Ratio of gallium content over the total Fe + Ga deposit as a function of concentration of Fe^{2+} ions in the co-deposition electrolyte system as measured by EDX.

of the Ga^{3+} ions relative to the Fe^{2+} ions in the co-deposition electrolyte system [5]. In this article, a further reduction of the concentration of the Fe^{2+} ions is reported for the plating of large structures, such as the millimetre size cantilevers described later.

The concentration of Fe^{2+} ions in the plating bath has a dominant effect on the Ga:Fe ratio of the resultant solid deposit. As shown in Fig. 1 the reduction of the concentration of Fe^{2+} ions and the increase of the pH result in a significant increase of the gallium content in the deposit. An appreciable amount of gallium deposit is only achieved when the Fe^{2+} ions concentration is reduced to 1/5 (0.006 M) or below of the recipe suggested by Iselt et al. [5]. Secondly, the suggested pH value of slightly less than 3.0 from the literature does not allow significant gallium deposition until the Fe^{2+} ions concentration is reduced to 1/10 (0.003 M) of the original recipe. Our findings indicate that there is a threshold around pH 3.4 for gallium deposition using the given co-deposition electrolyte system.

Only when the pH is 4.0 can a homogenous film be deposited across a large area substrate with a length of a few centimetres. Due to the competitive nature between the Ga^{3+} and Fe^{2+} deposition mechanisms, any variations in the uniformity of either the rate of diffusion of the chemical species arriving at the surface of the substrate or of the current density across the substrate, would create variations in the Ga:Fe deposition ratio.

As quantified by Element Dispersive X-Ray (EDX) elemental analysis, a $\text{Ga}_{0.19}\text{Fe}_{0.81}$ film was achieved for a pH of 4.0 and 0.006 M Fe^{2+} concentration. This Ga:Fe ratio is close to that required for optimal magnetostrictive behaviour [13]. Further reduction of the Fe^{2+} to 0.003 M Fe^{2+} concentration resulted in the deposition of a $\text{Ga}_{0.29}\text{Fe}_{0.71}$ film. A separate EDX element mapping was carried out on a cross-sectional view of the $\text{Ga}_{0.29}\text{Fe}_{0.71}$ sample as shown in Fig. 2. The results obtained for $\text{Ga}_{0.19}\text{Fe}_{0.81}$ were found to be similar to those obtained for the $\text{Ga}_{0.29}\text{Fe}_{0.71}$ sample. The preparation of the film was performed using Broadband Ion Beam (BIB) machining by the Company MCS Ltd [14], which allows deformation and smear-free cross-sectioning without obscuring the sample surface. The process uses a BIB system, which allows representative sample sizes of up to 2 mm to be cross-sectioned without mechanically touching the sample. This is a significant step change, which enables absolute confidence that deformation of the device did not occur during preparation for analysis. The samples were analysed using a combination of Field Emission Scanning Electron Microscopy (FESEM) and EDX. The individual element maps of Ga and Fe indicate no gradient in Ga content and that the Ga is evenly distributed within Fe–Ga alloy film. There is also no oxygen content detectable by the EDX, meaning that no gallium hydroxide was incorporated into the deposit during the electrochemical deposition. The cross-section of an oxygen-free, dense $\text{Ga}_{0.29}\text{Fe}_{0.71}$ electrodeposited thin

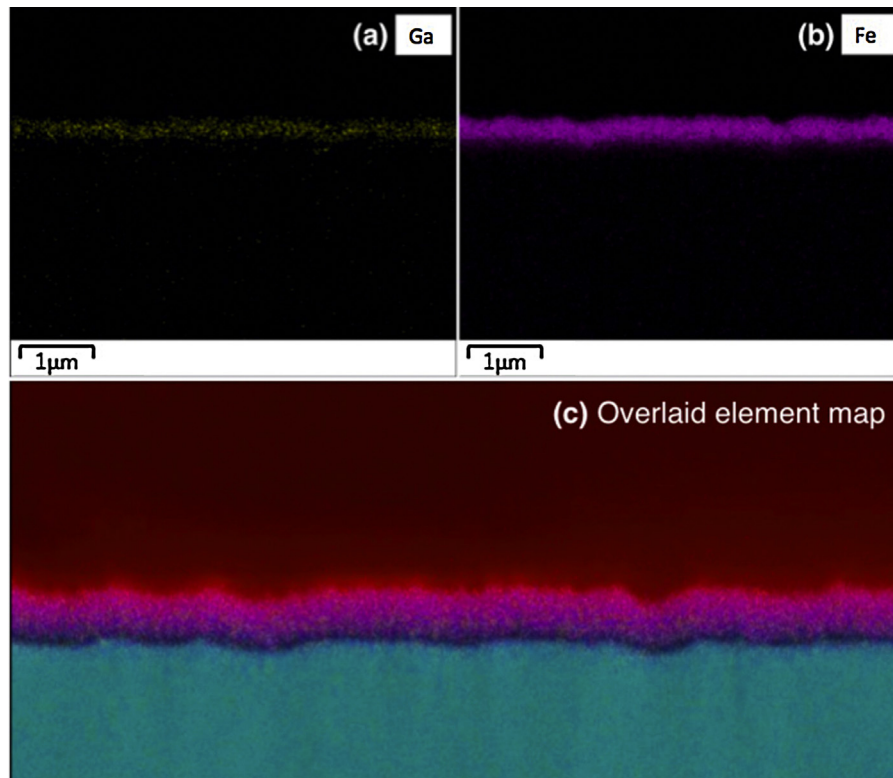


Fig. 2. EDX Ga, Fe and overlaid element maps on a cross-sectional overview.

film is imaged by the FESEM as shown in Fig. 3 (top image) and found to have an average film thickness of approximately 600 nm after 115 plating cycles. The interface between the alloy and the substrate seems more interstitial as observed in Fig. 3 (bottom image) in the case of a sample with only Fe electrodeposited from the co-deposition electrolyte system.

FESEM images from the surface of the deposited film also indicate that homogeneous deposition of the alloy took place as no separate Ga and Fe aggregate domains can be observed for both the $\text{Ga}_{0.19}\text{Fe}_{0.81}$ and the $\text{Ga}_{0.29}\text{Fe}_{0.71}$ samples as shown in Fig. 4.

3. Measurement of the magnetostrictive effect

A 20 μm thick copper foil was plated with a 1 μm thick $\text{Ga}_{0.19}\text{Fe}_{0.81}$ film at pH 4.0. Then the foil was used as a substrate for cutting 5 mm long and 1 mm wide cantilevers, as those shown in Fig. 5. The cutting process was performed using a frequency-doubled diode-pumped Yb:YVO₄ laser (Spectra Physics Lasers, Inc.) which provided 65 ns pulses with a 30 kHz pulse repetition frequency at a 532 nm wavelength. The cutting of cantilevers was performed by moving a focused laser beam across the foil at a 20 mm s⁻¹ velocity using a galvo-scanner, which was equipped with a 108 mm focal length flat-field (T-theta) lens to provide an approximately 16 μm diameter spot on the foil. The pulse energy used for cutting was 130 μJ corresponding to a 3.9 W average power, whilst the cutting tolerance was better than ±20 μm.

A magnetostrictive material such as Galfenol always strains positively in a magnetic field irrespective of the field direction. If the film is deposited on the top surface then the cantilever will bend downwards in the presence of a parallel magnetic field. This was found to be the case. Since the deflection was small compared to the cantilever length and of the same magnitude irrespective the parallel field direction the magnetostatic forces were assumed to be negligible.

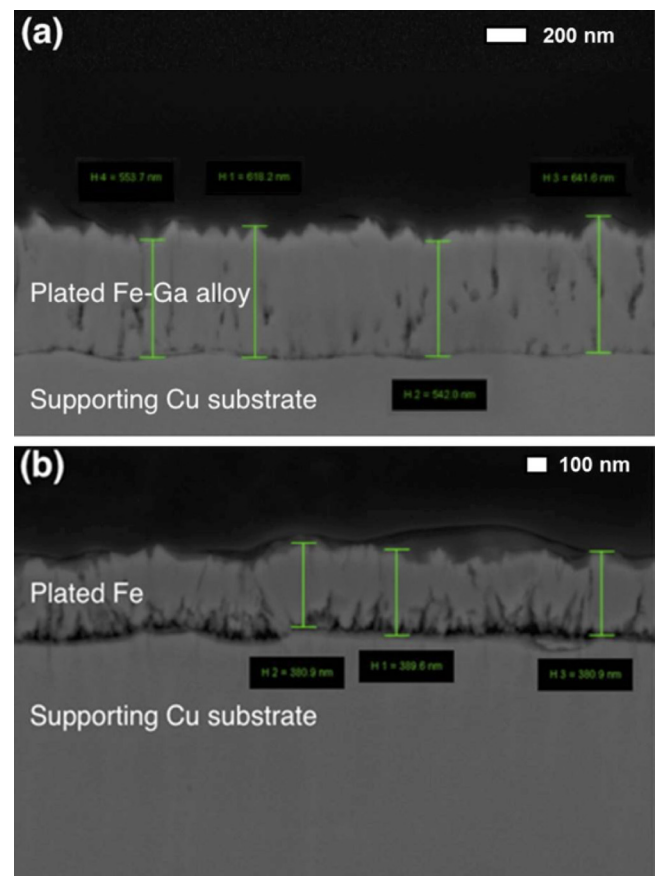


Fig. 3. Cross-sectional FESEM images of top the electroplated $\text{Ga}_{0.29}\text{Fe}_{0.71}$ alloy and bottom the electroplated Fe film with no detectable Ga.

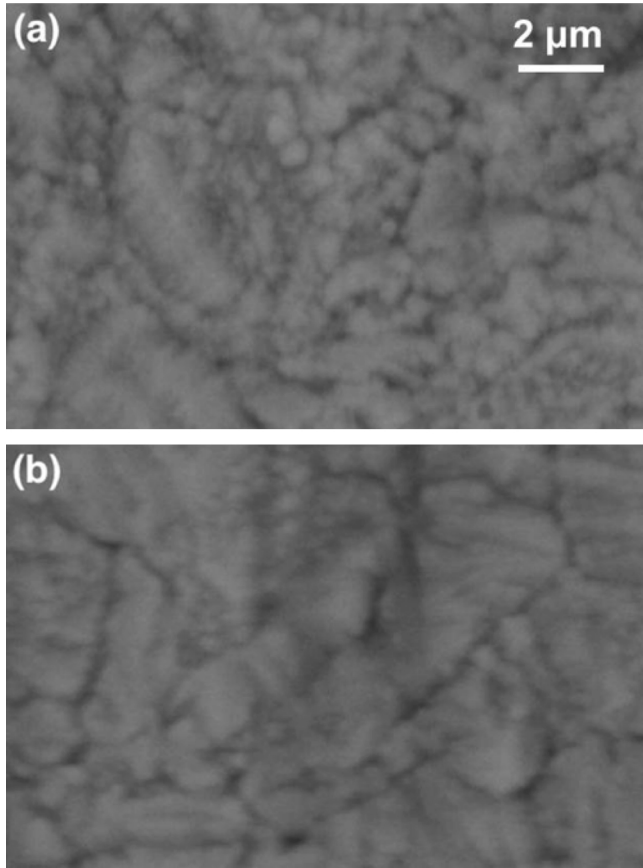


Fig. 4. Top view FESEM images of (a) $\text{Ga}_{0.19}\text{Fe}_{0.81}$ and (b) $\text{Ga}_{0.29}\text{Fe}_{0.71}$ electrodeposited films.

3.1. Test setup

A C-core used power transformer, Wiltan type 10/8/13, was machined to produce a 15 mm gap between the pole faces [15]. Eighty-eight turns of 1 mm^2 copper wire was wound on the core to provide up to 60 kA/m between the pole faces. The experimental setup is shown in Fig. 6. Vertical deflection was measured using a Micro Epsilon NCDT2400 confocal displacement probe [16], denoted by the label A in the figure. The field intensity was measured at the cantilever end. The output was averaged over 5 measurements and sampled at 300 Hz, which enabled the displacement to be measured within $\pm 50 \text{ nm}$. Background vibrations were minimised by bolting the rig to a Newport mini optical table. Current was switched using a type 20N06 MOSFET via and

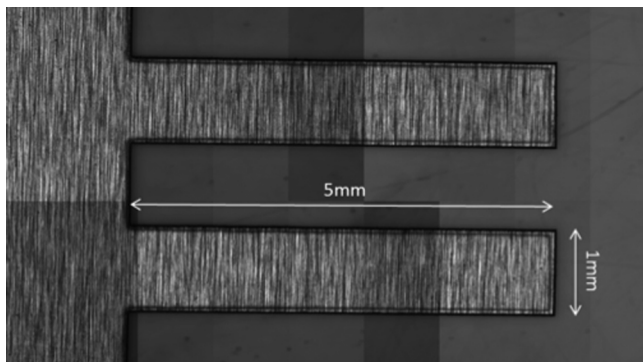


Fig. 5. Optical microscope image of the laser cut cantilevers.

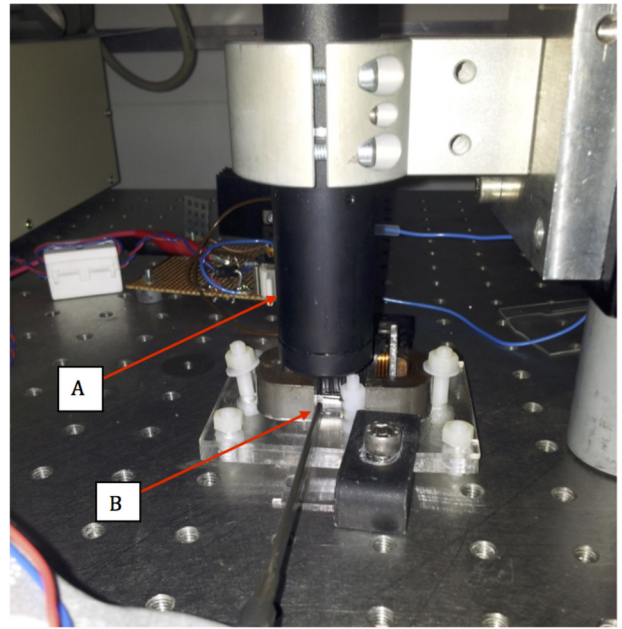


Fig. 6. Experimental setup for the measurement of the deflection of the cantilever beam.

Agilent 33220A pulse generator. The field between the pole faces was measured by a Hirst flux probe, located between the pole faces and labelled B.

3.2. Modelling of the strain in the Galfenol film

The cantilevers are modelled as a beam with one simply supported end and the other end free. The beam is constructed of two materials of different Young's modulus and thickness as shown in Fig. 7. The strain in the Galfenol film, λ , can be calculated using Eq. (1) [5]:

$$\lambda = \frac{(1 + \nu_g) \cdot (E_{\text{Cu}} \cdot t^2) \cdot D}{3L^2 \cdot E_g \cdot d \cdot (1 - \nu_{\text{Cu}})} \quad (1)$$

where E_g and E_{Cu} are the Young's moduli of Galfenol and copper, respectively and t is the thickness of substrate, here the copper foil. D is the deflection of the beam, d is the Galfenol film thickness and ν_g and ν_{Cu} are the Poisson's ratios of Galfenol film and copper, respectively. L is the length of the cantilever. The values of E_g were taken from [4] and values of E_{Cu} and ν_{Cu} were taken from [5].

3.3. Experimental results

With the beams mounted in the test jig shown in Fig. 7, pulse fields were applied onto the cantilevers with the confocal probe focused on each beam in turn. Fig. 8 shows a typical deflection

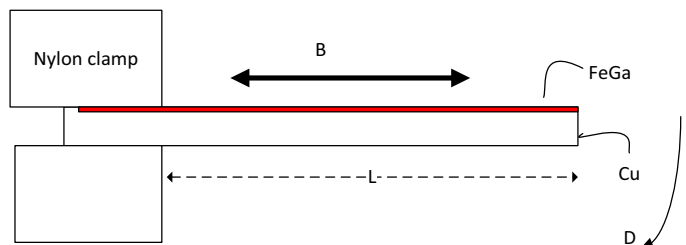


Fig. 7. Schematic of the cantilever of length L . B is the applied field with the arrows indicating direction. FeGa is the Galfenol film. In this configuration the deflection D is always downwards if the film is magnetostrictive.

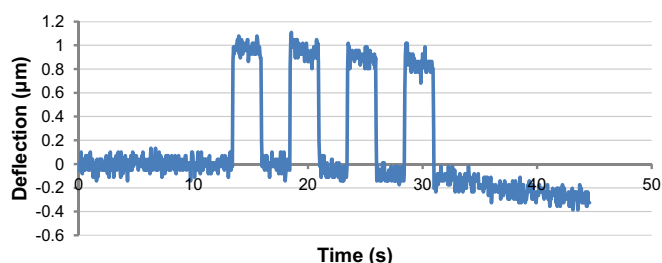


Fig. 8. Beam deflection in μm as a function of time with a 40 mT (31 kA/m) applied field with duty cycle of 50%.

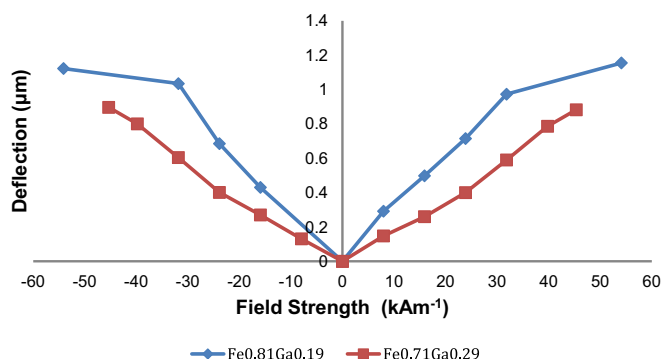


Fig. 9. Deflection in microns of the cantilever beams as a function of the field strength for $\text{Fe}_{0.81}\text{Ga}_{0.19}$ and $\text{Fe}_{0.71}\text{Ga}_{0.29}$ samples. The vertical error bars at ± 1 standard deviation are about 50 nm.

response to a 0.2 Hz applied pulse. It should be noted that the sagging of the cantilever over time is due to the relaxation of the plated film stress.

Measurement of the deflection of the $\text{Fe}_{0.81}\text{Ga}_{0.19}$ and $\text{Fe}_{0.71}\text{Ga}_{0.29}$ coated cantilevers as a function of the magnetic field strength was carried out and is displayed in Fig. 9. The samples display the characteristic V-shape curve with the material saturating at fields above 38 kA/m for the $\text{Fe}_{0.81}\text{Ga}_{0.19}$ sample, and the $\text{Fe}_{0.71}\text{Ga}_{0.29}$. Because the orientation of the crystal lattice, as determined by XRD analysis was inconclusive, the strain was calculated at maximum deflection. Using Eq. (1), the $\text{Fe}_{0.81}\text{Ga}_{0.19}$ film demonstrated a magnetostrictive coefficient of 96 ppm for a deflection of 1.15 μm with $E_{\text{Cu}} = 111$ GPa, and $E_{\text{g}} = 70$ GPa for an average thickness of 0.6 μm and 20 μm for the Galfenol and copper layers, respectively. Measurements seen in the top of Fig. 3 indicate however that thickness can vary between 553 and 641 nm. This compares with results obtained in [5], for which a value of λ of 109–147 ppm with sputtered films oriented in the 100 direction for Gallium concentration of 19% and the results obtained in [8], where a value of 140 ppm for a 148 nm thick electrodeposited $\text{Fe}_{83}\text{Ga}_{17}$ film was measured using a different methodology. The strain for $\text{Fe}_{0.79}\text{Ga}_{0.21}$ has been calculated at 75 ppm.

The lower values seen here are due to the uncontrolled lattice orientation. By comparison, Galfenol films grown by melt extrusion have a coefficient ranging from 250 to 400 ppm [5]. It is worth noting that, in our case, the cantilever beam exhibited a small amount of upward curling indicating the presence of tensile stress in the deposited film.

4. Conclusions

This paper reports a technique for the electrochemical deposition of $\text{Fe}_{0.81}\text{Ga}_{0.19}$ and $\text{Fe}_{0.71}\text{Ga}_{0.29}$ films that exhibit promising magnetostriction characteristics. An electroplating process was

developed that enables the deposition of solid and consistent films close to the ideal composition of 80:20 iron/gallium concentration with high tolerance towards current density variations, making it ideal for the electrodeposition of micropatterned structures for MEMS devices. A first potential magnetostrictive application is demonstrated in the form of bilayer cantilever beams that deflect in response to an external bias magnetic field. A 600 nm thick Ga–Fe alloy film was grown on top of a 20 μm thick copper foil after 115 plating cycles and subsequently cut to form millimetre-size cantilever beams. The deflection of the beams in response to an external magnetic excitation supplied by a custom-built test rig was monitored. A strain coefficient at maximum deflection was measured to be $\lambda = 96$ ppm. The observed magnetostrictive properties represent a promising result for further application into more complex MEMS and millimetre scale devices such as magnetic field sensors or magnetic actuators.

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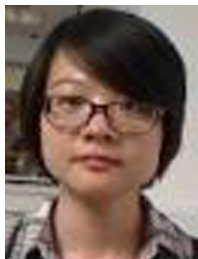
Biographies



Jack H.-G. Ng graduated from Heriot-Watt University with a Ph.D. in simplifying metals fabrication for micro-engineering and is the first inventor of a patent of an artificial photosynthesis-inspired manufacturing process. Following his further post-doctorate positions in fabrication and packaging of sensors at the applied chemistry level, he has been awarded by The Royal Society of Edinburgh and Scottish Enterprise as an Enterprise Fellow currently commercialising the antimicrobial property of copper in order to reduce infections in the healthcare environment.



Paul M. Record graduated from Strathclyde University with a Ph.D. and has worked on biomedical instrumentation for visual prosthesis at University of Birmingham and electrical impedance imaging at University of Keele. Later work included instrumentation for satellite relay data loggers for phocids and cetaceans in the open ocean at University of St. Andrews. Work on implanted RF linked tags for these animals lead to an interest in RFIDs and embedded antennas in the 10 MHz to 2 GHz bands. Coupled work on sensors included magnetostrictive materials for wireless sensors. He has three patents on antennas, electric field sensors and tag synchronisation.



Xinxin Shang graduated from Heriot-Watt University in 2013 with an M.Sc. degree in materials for sustainable and renewable energies.



Krystian L. Włodarczyk graduated from the Wrocław University of Technology (Poland) in 2006 with an M.Sc. degree in electronics and telecommunications. Then, he joined the Lasers and Photonics Applications group at Heriot-Watt University as a PhD student. Krystian's Ph.D. research studies involved CO₂ laser micro-machining of optical glasses and investigating new applications for CO₂ laser polishing. He graduated with a Ph.D. degree in June 2011. Currently, Krystian is a Research Associate within the High Power Laser Application group at Heriot-Watt University where he works on various projects involving laser processing of metals and optical glasses.



Duncan P. Hand has been a member of staff at Heriot-Watt University for over 20 years, following a PhD in fibre optics at the University of Southampton. The focus of his research activity is applications of high power lasers, in particular in manufacturing, and he is Director of the EPSRC Centre for Innovative Manufacturing in Laser-based Production Processes, a 5-university Centre. Other research focuses on delivery of high peak power laser light through novel microstructured optical fibres (with Bath University), with applications in manufacturing and medicine. He is also currently Director of Research and Deputy Head of the School of Engineering and Physical Sciences.



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applications.

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